

PATENT
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THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants:	Burns, et al.)	Examiner:	Peter Chin
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Appl. No.:	09/688,332)	Art Unit/T.C.:	1731
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Filed:	October 10, 2000)	Customer No.:	04-1403
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Title:	Soft Paper Products with)	Confirmation No.:	1223
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DECLARATION UNDER 37 C.F.R. § 1.132

We, Werner F.W. Lonsky and Alberto R. Negri, do hereby declare as follows:

1. We are two of the six co-inventors of the claimed invention described and defined in the above-captioned United States patent application.

2. I, Werner F.W. Lonsky, have a home address of 34 Crestview Court, Appleton, Wisconsin 54915. I have a doctorate in organic chemistry from the University of Vienna. I have been employed by Kimberly-Clark Worldwide, Inc. for 18 years, in the Pulp Development Department for 9 years and in the Fiber Technology Department for 9 years.

3. I, Alberto R. Negri, have a home address of 1127 East Capitol Drive, Appleton, Wisconsin 54911. I have a bachelor's degree in chemistry and chemical engineering, and I have a Masters of Science and a Ph.D. in polymer chemistry from the University of Washington. I have been employed by Kimberly-Clark Worldwide, Inc. in the Fiber Technology Department for 6 years.

4. Independent claim 82 of our patent application, for instance, is directed to a method for forming a paper web that contains a first layer formed primarily from hardwood fibers. The hardwood fibers in this first layer are treated with a first hydrolytic

enzyme to hydrolyze the hardwood fibers and form aldehyde groups predominantly on the surface thereof. The dosage of this hydrolytic enzyme is from about 0.1 to about 10 s.e.u. (standard enzyme units) per gram of oven-dried pulp. The first hydrolytic enzyme comprises a cellulose-binding domain free endo-glucanase (hereinafter, "CBD-free endo-glucanase"). The hardwood fibers are further treated with a cross-linking agent that forms a bond with the aldehyde groups on the surface of the hardwood fibers.

5. In a Final Office Action mailed May 15, 2003, the claims of our patent application were rejected in view of International Patent Publication No. WO 98/56981 entitled "Modified Cellulosic Fibers and Fibrous Webs Containing These Fibers" to Seger, et al. We are familiar with this publication.

6. The Seger, et al. publication is directed to modified cellulosic fibers having a reduced dry zero span tensile index. To obtain the modified cellulosic fibers, a cellulase-containing enzyme is added to an aqueous slurry of fibers. (Page 13). The enzyme modifies the morphology of the fibers. (Page 13). After mixing of the fibers and enzyme preparation, the mixture is preferably, though not necessarily, combined with a debonder or chemical softener to preserve the fiber morphology modifications that result from enzymatic action. (Page 13). Specifically, Seger, et al. indicates that the addition of a debonder to wet enzyme-modified fibers prevents the "repair" of the fibers that would otherwise take place upon drying. (Page 17).

7. As requested by the Patent Examiner, we conducted an experiment to compare hardwood fibers treated with a CBD-free endo-glucanase enzyme to hardwood fibers treated with one of the endo-glucanase enzymes used in Seger, et al. We modeled our experiment after Example 4, Samples "Control Euc" and "4A" of Seger, et al. (described at pages 50-53), since this Example involved enzyme treatment of hardwood fibers (specifically, eucalyptus fibers).

8. First, a furnish of VCP eucalyptus dry-lap pulp was formed. The eucalyptus pulp was reslushed in a 20-Liter hydropulper, dewatered, fluffed, and kept under refrigeration. The consistency of this eucalyptus pulp was about 41%. This furnish of eucalyptus pulp was used to prepare four 30-gram samples of oven-dry pulp, which were labeled Samples 1-4. These four samples were continuously agitated using

a Lightnin'® mixer. (Pages 30-31 of Seger, et al. similarly describe that the samples tested in their Examples began as 30-gram samples of oven-dry pulp.)

9. Sample 1 was the control pulp sample in this experiment. Sample 1 included the VCP eucalyptus pulp described in the above paragraph with no enzyme treatment. Sample 1 underwent 1 hour of mixing using the Lightnin'® mixer. The fiber consistency of the pulp of Sample 1 was about 3%, and the 1-hour mixing took place at a temperature of about 120°F (about 49°C) using the Lightnin'® mixer (rheostat at 100 units in a scale of 0-135). Sample 1 was treated with a hypochlorite solution (100 ppm) at the end of the mixing time. Sample 1 was then washed, dewatered and kept overnight under refrigeration.

10. Sample 2 was the pulp sample modified with one of the endo-glucanase enzymes disclosed by Seger, et al. Specifically, Sample 2 included VCP eucalyptus pulp which underwent 1 hour of treatment with a solution of Carezyme®, one of the endo-glucanase enzymes of Seger, et al., and the Carezyme® was added to the pulp of Sample 2 in an amount such that the activity level of the enzyme in Sample 2 was 50 Cellulase Viscosity Units per gram of oven-dry pulp (CEVU/godp).¹ (Page 10, lines 13-14 of our patent application explain that the amount of enzyme administered can be denoted in terms of its activity per mass of dry pulp.) The fiber consistency of the pulp of Sample 2 was about 3%, and the 1-hour enzyme treatment took place at a temperature of about 120°F (about 49°C) using the Lightnin'® mixer (rheostat at 100 units in a scale of 0-135). At the end of the treatment time, enzyme activity was

¹ In Example 4, Sample 4A of Seger, et al., a commercially available liquid form of Carezyme® was used, which is described at page 12 of Seger, et al. as having an enzyme activity of 5000 Cellulase Viscosity Units per gram (CEVU/g). In Seger, et al., this liquid form of Carezyme®, having an enzyme activity of 5000 CEVU/g, was made into a 1% solution, meaning that the treatment solution had an enzyme activity level of 50 CEVU/g. 30 mL of that solution were then used to treat the 30-gram eucalyptus pulp sample that became Sample 4A of Seger, et al., such that the resulting enzyme activity in Sample 4A was 50 CEVU/gram of oven-dry pulp. In our experiment, we used analytical grade Carezyme® powder (Novo Nordisk batch 17-1196), which was readily available in our lab. This powder form of Carezyme® has an enzyme activity of 4931 CEVU/g. To prepare our Carezyme® solution, 1.01 grams of this Carezyme® powder were dissolved in 100 mL of distilled water to make a 1% treatment solution having an enzyme activity of 50 CEVU/g. 30 mL of this solution were then used to treat Sample 2, a 30-gram eucalyptus pulp sample, such that the resulting enzyme activity in Sample 2 was 50 CEVU/gram of oven-dry pulp. Thus, the enzyme activity level of 50 CEVU/gram of oven-dry pulp used in Sample 2 above mirrors the enzyme activity level used for Sample 4A of Seger, et al.

destroyed with a hypochlorite solution (100 ppm). Sample 2 was then washed, dewatered and kept overnight under refrigeration.

11. Sample 3 was the first pulp sample modified with a CBD-free endoglucanase enzyme. Specifically, Sample 3 included VCP eucalyptus pulp which underwent 1 hour of treatment with a solution of a CBD-free enzyme, Novozyme® SP-988, which was used in the Examples at pages 24-36 of our patent application. Novozyme® SP-988 was added to the pulp of Sample 3 in an amount such that the activity level of the enzyme in Sample 3 was the same as the activity level of the enzyme in Sample 2 (i.e., 50 CEVU/gram of oven-dry pulp, which is equivalent to 1.8 standard enzyme units (s.e.u.) per gram of oven-dry pulp, where s.e.u.'s are the units by which the activity level of Novozyme® SP-988 is expressed).² Again, the fiber consistency of the pulp of Sample 3 was about 3%, and the 1-hour treatment took place at a temperature of about 120°F (about 49°C) using the Lightnin'® mixer (rheostat at 100 units in a scale of 0-135). At the end of the treatment time, enzyme activity was destroyed with a hypochlorite solution (100 ppm). Sample 3 was then washed, dewatered and kept overnight under refrigeration.

12. Sample 4 was prepared exactly like Sample 3 above, except that the VCP eucalyptus pulp only underwent 20 minutes of treatment with the Novozyme® SP-988 enzyme (rather than 1 hour of treatment).

13. Four handsheet sets having basis weights of about 60 grams per square meter³ were then made under identical conditions according to the following procedure,

² Page 10, lines 14-26 of our patent application describe what is meant by a dosage of one standard enzyme unit (s.e.u.) of an endo-glucanase enzyme, and dosages expressed in s.e.u./gram of oven-dry pulp are used in the claims of our application.

³ The Examples of Seger, et al. use low-density handsheets made according to TAPPI Standard Procedure T205 (with some modifications) having a target basis weight of 26.8 gsm (16.5 pounds per 3,000 square feet) and a target density of about 0.15 grams/cc. (Seger, et al., pages 23-25). The 60 gsm handsheets used in our experiment are higher in basis weight and lower in bulk when compared to handsheets tested in the Examples of Seger, et al. because our 60 gsm handsheets were formed using low pressure wet pressing (a required step in the standard procedure used to make these 60 gsm handsheets). It should be noted that all of the properties tested and reported in Table 1 below (specifically, dry zero span tensile strength, tensile index, and tear index) are basis weight independent. Furthermore, it is known that lighter handsheets (i.e., handsheets having a basis weight less than 60 gsm) will actually raise the zero breaking length appreciably. See TAPPI Standard Procedure T-231 cm-96, Additional Information, Item 16.5.

which is described in Example 3 at page 34 of our patent application. Handsheet 1 was prepared by diluting fiber Sample 1 in water to a consistency of 1.2 weight percent in a British Pulp Disintegrator and allowing the dispersed sample to soak for 5 minutes. The sample was then pulped for 5 minutes at ambient temperature, diluted to 0.3 percent consistency and formed into a handsheet on a square (9 x 9 inches) commercially available as the Valley Handsheet Mold (Voith Inc., Appleton, Wis.). The handsheet is couched off the mold by hand using a blotter and pressed wire-side up at 100 pounds per square inch for 1 minute. Then the handsheet was dried wire-side up for 2 minutes to absolute dryness using a Valley Steam Hotplate (Voith Inc., Appleton, Wis.) and a standard weighted canvas cover having a lead-filled (4.75 pounds) brass tube at one end to maintain uniform tension. The resulting handsheet was then conditioned in a TAPPI lab environment (a humidity-controlled room at 23°C, 50% relative humidity) prior to testing. Handsheets 2-4, using fiber Samples 2-4 respectively, were formed and conditioned in exactly the same manner.

14. Handsheets 1-4 were tested for their (1) dry zero span tensile strength, (2) tensile index, and (3) tear index. The dry zero span breaking strength of the handsheets was tested according to TAPPI Standard Procedure T-231 cm-96. This testing employed a Pulmac TroubleShooter having a jaw width of 15 mm and a clamping pressure of 80 psi. The sample strips tested for dry zero span tensile strength were 25 mm wide. The testing was performed similarly to the procedure described at page 26 of Seger, et al. The tensile index of Handsheets 1-4 was measured according to TAPPI Standard Procedure T220 sp-96 and similarly to the dry tensile strength index testing described at page 26 of Seger, et al. Specifically, this testing employed a tensile tester (MTS Alliance RT/1) using 1-inch wide strips. The tear strength of Handsheets 1-4 was tested according to TAPPI Standard Procedure T414 om-88. This testing employed a TMI Monitor/Tear Model 83-11-00, calibrated with 200 gram weight, using 63-mm wide strips.

15. The results of such testing were obtained and are shown in Table 1 below:

Table 1

	Handsheet 1	Handsheet 2	Handsheet 3	Handsheet 4
Enzyme Used to Treat VCP Eucalyptus Pulp	None (Control)	Carezyme® (Seger, et al.)	Novozyme® SP-988 (1 hr.) (Invention)	Novozyme® SP-988 (20 min.) (Invention)
Dry Zero Span Tensile Strength (DZST) (N)	83.22	58.75	79.22	81.23
DZST Standard Deviation	±5.48	±5.82	±5.71	±4.75
Change in DZST (%) Compared to Handsheet 1	--	-29.4%	-4.8%	-2.4%
Tensile Index (N·m/g)	12.87	11.71	16.18	15.63
Tensile Index Standard Deviation	±0.53	±0.43	±0.19	±0.65
Change in Tensile Index (%) Compared to Handsheet 1	--	-9.0%	+25.7%	+21.4%
Average Tear Index (mN·m²/g)	3.67	2.30	3.48	3.60
Tear Index Standard Deviation	±0.25	±0.12	±0.09	±0.22
Change in Tear Index (%) Compared to Handsheet 1	--	-37.4%	-5.0%	-2.0%

16. The results in Table 1 above show that the reduction in DZST strength for the fibers treated with Carezyme® in Handsheet 2 is far more pronounced (almost 30%) when compared with the DZST reductions for the fibers treated with the CBD-free endoglucanase, Novozyme® SP-988, in Handsheets 3 and 4. The very small (i.e., 4.8% and 2.4%) reductions in DZST observed for Handsheets 3 and 4 may have been due to the fact that our mixing conditions (i.e., using the Lightnin'® mixer) were very different from the high shear conditions one might expect in a hydropulper. Yet, there is virtually no statistical difference for the DZST values for Handsheets 3 and 4 when compared with Control Handsheet 1, since the DZST reductions of 4.8 and 2.4 are below the respective standard deviations.

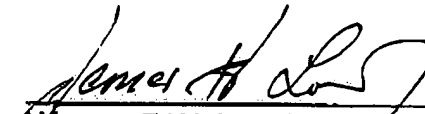
17. The results in Table 1 above further show pronounced increases in tensile index for Handsheets 3 and 4 (specifically, +25.7% and +21.4%) compared to Control Handsheet 1, while the tensile index for Handsheet 2 (treated with Carezyme®) decreased 9% compared to Control Handsheet 1.

18. These results also show that the reduction in tear index for Handsheet 2, treated with Carezyme®, is far more pronounced (37.4%) when compared to the reductions in tear index (5.0% and 2.0%) for Handsheets 3 and 4, which contained eucalyptus fibers treated with Novozyme® SP-988. Again, the very small reductions in DZST observed for Handsheets 3 and 4 may have been due to the fact that our mixing conditions (i.e., using the Lightnin'® mixer) were very different from the high shear conditions one might expect in a hydropulper. Also, the tear index reduction for Handsheet 4 is statistically insignificant, since the tear index reduction of 2.0% is below the respective standard deviation for Handsheet 4.

19. The results of this experiment generally indicate differences observed when hardwood fibers are treated with a cellulose-binding domain free endo-glucanase enzyme, as recited in the claims of our patent application, versus when hardwood fibers are treated with the CBD-containing endo-glucanase enzymes described by Seger, et al.

20. We further declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the above-captioned application or any patent issuing thereon.

November 17, 2003
Date



Werner F.W. Lonsky

November 17, 2003
Date



Alberto R. Negri